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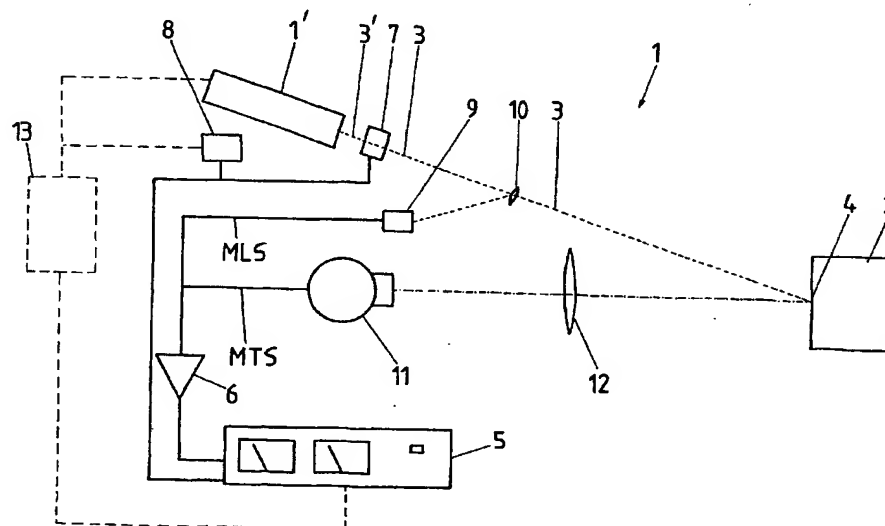
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(54) Title: METHOD AND SYSTEM FOR MEASURING THE THERMAL DIFFUSIVITY



(57) **Abstract:** The present invention concerns a method and a system for measuring the thermal diffusivity. Said method comprises the steps of: - subjecting a surface region of an object to a modulated heating beam (3) while providing a signal (MLS) thereof, the heated spot or area (4) of the surface of the sample (2) having a definitive diameter and a fixed intensity distribution profile; - providing a signal (MTS) proportional to the temperature of the heated spot area (4); - determining the phase difference between the modulated beam signal (MLS) and the resulting modulated temperature signal (MTS); - using said determined phase difference and the associated frequency to work out the thermal diffusivity value of said object (2).

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METHOD AND SYSTEM FOR MEASURING THE THERMAL DIFFUSIVITY

The present invention is related to the measurement of physical properties of materials, components or articles, and concerns a method for measuring the thermal diffusivity and a corresponding system able to perform such a measurement.

5 The thermal diffusivity is a material thermal property which is important in many engineering applications.

Various methods are already known in order to perform such measurements, the two most efficient and precise ones using laser based techniques.

10 Thus, a first known method of this type is the so-called "Laser flash" method.

In this latter method, a sample of the material to be investigated must be prepared. Furthermore, two opposite sides of said sample must be parallel and its thickness known with high precision.

15 The sample is then uniformly heated by a laser pulse on one side. On the opposite side of this sample an IR detector measures the temperature rise. The rise time of the temperature is linked to the thermal diffusivity which can thus be determined.

20 Nevertheless, this first known method shows a number of disadvantages and restrictions when it is practically implemented, among which one can cite: the measurement can not be performed on a bulk; a sample must be extracted and accurately prepared; two faces of the sample need to be parallel and accessible.

25 A second known method of the aforementioned type is the so-called "3D-Photothermal radiometry".

30 In this second method, the sample is heated by a focused modulated laser beam and on the same side of the sample an infrared detector (IR-detector) measures the modulated surface temperature at an increasing distance from the heated spot. The phase difference between the modulated laser and the modulated temperature depends on this distance and on the thermal diffusivity of the material.

However, this latter method does also present certain disadvantages in connection with its practical implementation, as the laser spot or the IR-detector must be moved, as the IR and visible optic need an

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extremely accurate preparation, and as the IR signal becomes rapidly very weak as the distance from the heated spot increases.

The main object of the present invention is to overcome at least some and preferably all of the above mentioned disadvantages and
5 restrictions.

Therefore, the present invention provides a method for measuring the thermal diffusivity characterized in that it comprises the steps of:

- 10 - subjecting a surface region of the object, whose thermal diffusivity has to be determined, to a modulated laser beam or to the modulated beam of a similar heating source while providing a signal thereof by, for example, measuring said beam and providing a signal proportional to the beam intensity, the heated spot or area of the surface of the object having a definitive diameter and a fixed intensity distribution profile;
- 15 - providing a signal proportional to the temperature on the heated spot or area;
- determining the phase difference or phase shift between the modulated beam signal and the resulting modulated temperature signal;
- 20 - using said determined phase difference or shift and its associated modulation frequency to work out the thermal diffusivity value of said object.

Providing a signal of the modulated beam applied to the surface region does not necessarily mean to measure said beam itself. For example, a small part of the beam can be deviated and a signal proportional to said
25 beam extracted from it.

Furthermore, it is neither necessary to measure the temperature of the heated spot or area. It is enough to provide a signal proportional to said temperature or with the same frequency and phase than said temperature.

- 30 The invention takes advantage of the fact that:
- given a fixed spatial distribution of an amplitude-modulated beam, e.g. a laser beam, heating the flat surface of an object;
 - 35 - given a fixed spatial sensitivity distribution of a surface temperature measurement system, e.g. infrared detector, providing a temperature signal;

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the phase lag of the temperature signal with respect to beam modulation signal depends only on the diffusion length of the heated material.

On two different materials with respective diffusivities α_1 and α_2 , the same phase lag is obtained at two different modulation frequencies f_1 and f_2 such that the corresponding diffusion lengths μ_1 and μ_2 are equal. Knowing that $\mu^2 = \alpha / (\pi f)$, where α is the diffusivity, it follows that $\alpha_1 / f_1 = \alpha_2 / f_2$.

The phase lag being measured on a reference material with well-known diffusivity, the diffusivity of the second material is retrieved from the above-mentioned formula.

The above-mentioned technique does not require the knowledge of the sensitivity and beam spatial distributions but requires a reference material. Alternatively, if the distributions are known, the absolute diffusion length can be theoretically determined measuring the phase lag at one or multiple modulation frequencies, without needing a reference material.

Since the measurement can be performed on a bulk, there is no need of extracting a sample. The measurement can be directly performed on a surface point of the object of interest. If one wants to measure the thermal diffusivity of a point on the surface, for instance, of a turbine blade, said blade is taken, put it in the right position in the measuring apparatus and the measure of its diffusivity on the desired point is performed.

One can also imagine that the measuring apparatus or system can be made so small and compact, that the system itself is taken (instead of the blade), put it in contact with the surface of the blade and the diffusivity of the desired point measured.

Advantageously, at least one modulation parameter can be subjected to controlled variation, preferably the frequency, which allows an accurate modulation and detection of said modulation.

In theory, if a semi-infinite solid is sinusoidally heated on a dimensionless surface point, the temperature of this point has a 0° phase shift with the heating source. On the other hand, if a semi-infinite solid is sinusoidally heated, in a homogeneous way on its surface, the phase shift with the heating source is -45° .

In practice, the surface is heated by a beam such as a laser with a given intensity distribution profile on a spot of finite dimension.

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Depending on the modulation frequency this situation can be similar to the point heating or to the homogeneous heating.

If the thermal diffusion length μ ($\mu = \sqrt{\alpha/\pi f}$, where α is the thermal diffusivity and f is the modulation frequency) is small with respect
5 to the diameter of the region heated by the beam, the situation is similar to the homogeneous heating.

If the thermal diffusion length μ is big, compared with the diameter of the region heated by the laser, the situation is similar to the point heating with a phase shift of 0° .

10 While the frequency is varied, the phase shift changes from 0° to -45° .

The knowledge of the intensity distribution profile of the laser beam Q and the knowledge of the sensitivity profile P of the temperature measuring system, for example an IR detection system (IR detector and its
15 lens), would allow to reckon the thermal diffusivity α , having measured the dependence of the phase shift $\Delta\phi$ from the modulation frequency f . The intensity distribution profile of the laser or heating beam and the sensitivity profile of the IR detecting system play the same role and only the profile, obtained with the convolution of the two, influences the measurement. If
20 the two profiles are changed, but their convolution remains the same, the measurement does not change. These two profiles are in general not well known and change if something in the measurement set-up is moved.

Since the object on which to perform the measurement is always placed in the same position and nothing moves during the
25 measurement, the two profiles Q and P remain always the same. The dependence of the phase shift from the modulation frequency can be recorded for a reference sample, with known thermal diffusivity α_r .

According to a simple and preferred embodiment of the invention, the thermal diffusivity value α_m of the analysed object is
30 evaluated by comparison with at least one reference or reference sample whose thermal diffusivity value α_r has been determined previously or is known and which has been subjected to the same measurement method in similar conditions of analysis.

Furthermore, the temperature is preferably, but not necessarily,
35 measured at the center of the heated spot or area, which allows to collect a stronger signal.

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Thus, the same measurement, as for the reference, is performed on the object of interest and the results compared with the ones obtained for the reference, in order to work out the value of thermal diffusivity α_m of said object. In this way, the knowledge of the two profiles Q and P is not
5 necessary. Therefore, they can remain unknown, but they must remain the same for the measurements of all objects to be analysed.

Let assume that the curve "phase shift vs. modulation frequency" has been recorded for a reference sample. The shape of this curve in a logarithmic frequency scale is a straight line. If, for instance, the
10 measurement is performed on an object and, for a certain modulation frequency of the heating source, a phase shift of -30° is measured, this phase shift is compared with the results obtained on the reference sample. Knowing the frequency at which the phase shift -30° was obtained for the reference sample, which has a known thermal diffusivity, the thermal
15 diffusivity for the object of the measurement can be worked out, knowing that, when the two phase shifts are the same: $\alpha_r/f_r = \alpha_m/f_m$ (1) where α is the thermal diffusivity, f the modulation frequency, "r" refers to "reference sample" and "m" refers to the object on which the measurement was performed.

20 In a first alternate carrying out of the said preferred embodiment, the modulation frequency of the heating beam is varied and the variation of the phase shift with respect to the heating source is recorded for a plurality of modulation frequencies. Since the resulting curve in a diagram with logarithmic frequency scale, is a straight line parallel to the
25 one obtained for the reference sample, one can obtain the value of thermal diffusivity by measuring the shifting required to superimpose the two curves.

In a second alternate carrying out of the said preferred embodiment, the modulation frequency is not varied and the phase shift is
30 recorded at a given frequency f_m . The dependence of the phase shift from the modulation frequency for the reference sample has been previously measured. The value of the modulation frequency f_r yielding the same phase shift for the reference is taken. Since the two phase shifts are the same, formula (1) allows to reckon the thermal diffusivity α_m of the object
35 to be measured knowing the thermal diffusivity α_m of the reference.

In a third alternate carrying out of the said preferred embodiment, the modulation frequency is varied till the phase shift reaches

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a predefined value obtained for the reference sample. Formula (1) is then applied to determine α_m .

In order to be able to perform measurements with high accuracy without needing accurately adjusted sensing and measuring means, the invention proposes advantageously that the modulated beam signal and the modulated temperature signal are both measured, successively and possibly repetitively, by preferably the same path and means, such as for example a lock-in amplifier preceded by a preamplifier, for example by masking alternatively the beam signal generator and the temperature signal generator. The used path and means add their own phase shift that is eliminated using the same path and means. If different measurement path and means are used, the inserted phase shift by the measurement system at each frequency should be known and subtracted.

It is worth to notice that the modulated beam signal at a given frequency is independent of the materials under test. Thus, if the instrumentation is stable enough, it can be measured once and memorized for future comparison with the temperature signal. In those conditions, the measurement procedure and the instrumentation might be simplified noticeably.

According to an advantageous feature of the invention, the provided heating beam is modulated by means of an acousto-optical modulator or a mechanical chopper, driven by an adjustable generator.

According to an other feature of the invention, the modulated beam signal is generated by a light sensor, such as for example a photodiode, receiving a deviated part of the beam, for example the light reflected by a lens through which the modulated beam is passing to be focused before striking the sample. The temperature signal is generated by an infrared sensor receiving the radiation sent out by the heated region or spot of the surface of the analysed sample and focused by an infrared lens.

The proposed method allows for thermal diffusivity measurement of a bulk material without the need of preparing a sample with a certain thickness and without the need of measuring the modulated surface temperature at different distances from the heated point. The method is therefore much simpler and versatile than the two aforementioned known methods and can be applied to objects with various surface geometry.

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The present invention also provides a system for measuring the thermal diffusivity of an object, said system comprising a laser device or a similar heating source, whose beam is directed towards a region or spot on the surface of said object.

5 Said system is characterized in that it also comprises means for modulating said laser or heating beam, means for generating a signal corresponding to said modulated laser or heating beam, means for generating a signal corresponding to the modulated temperature of the region or spot struck by the modulated beam and amplifying and
10 measuring means able to determine at least the phase difference or a shift between the modulated beam signal and the resulting modulated temperature signal and possibly the thermal diffusivity value of said object based upon said phase difference.

15 Preferably, said system is adapted to perform the thermal diffusivity measurement method as described before.

The present invention will be better understood thanks to the following description and drawing of an embodiment of said invention given as non limitative example thereof, the figure of said drawing consisting of a schematic and functional view of a system according to the
20 present invention.

As shown on the enclosed figure, the thermal diffusivity measurement system 1 mainly comprises a laser device or a similar heating source 1' whose beam 3' is directed towards a region or spot 4 on the surface of a sample 2 to be analysed. Said system 1 also comprises means 7,
25 8 for modulating said beam 3', a lens 10 for focusing the beam on a spot of desired diameter on the sample surface, a means 9 for generating a signal MLS corresponding to the modulated laser beam 3, means 11, 12 for generating a signal MTS corresponding to the modulated temperature of the region or spot 4 struck by the modulated beam 3 and amplifying and
30 measuring means 5, 6 able to determine at least the phase difference or shift between the modulated beam signal MLS and the resulting modulated temperature signal MTS and possibly the thermal diffusivity value α_m of said material sample 2 based upon said phase difference.

The sample 2 is preferably heated by a modulated laser beam 3
35 on a spot 4 of diameter D with fixed intensity distribution profile P. The IR-detector or sensor 11 measures the modulated temperature, preferably at the center of the heated spot 4, as the modulation frequency is varied.

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The phase difference between the modulated temperature and the modulated laser depends on the modulation frequency, the diameter D, the intensity distribution profile P and the thermal diffusivity. The heating beam 3' profile and the detector 11 sensitivity profile (i.e. the portion of the sample 2 surface whose emitted infrared radiation strikes the detector 11) play the same role. It follows that the heating spot 4 can be small and the sensitivity profile of detector 11 large.

In practice, the measurement can be performed, for example, by varying the modulation frequency till the phase difference reaches the same value than that obtained for the reference sample.

Since only one surface must be accessible, the measurement can be performed without sample preparation on a bulk. Moreover there is no need of any mechanical displacement of any component. All components of the system 1 remain in the same position and only the modulation frequency is varied.

According to a preferred featuring and implantation of the invention, the measuring means consist of a lock-in amplifier 5 preceded by a preamplifier 6, the provided laser beam 3' is modulated by means of an acousto-optical modulator 7, or a mechanical chopper, driven by an adjustable generator 8 and the modulated laser signal MLS is generated by a light sensor 9, such as a photodiode, receiving part of the light of the beam reflected by the lens 10 through which the modulated laser beam 3 is passing before striking the sample 2.

The temperature signal MTS is generated by an infrared sensor 11 receiving the radiation sent out by the heated region or spot 4 of the surface of the analysed sample 2 and focused and possibly filtered by an infrared lens 12.

As can be seen on the figure, both signals MLS and MTS are preferably, but not necessarily, handled by the same measuring path.

The lock-in amplifier measures the phase of the input signal with respect to a reference signal provided by the generator 8 driving the beam amplitude modulator. The input signal is the sum of the short circuit currents of the photodiode and the infrared detector. These currents are successively zeroed masking the light entering in the photodiode or the infrared radiation entering in the detector. The difference of the measured phases is the phase shift between beam amplitude signal and the temperature signal.

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As mentioned before, in case of enough stable instrumentation, the phase of the photodiode signal can be measured once in factory and memorized for future comparison with infrared detector signal, simplifying noticeably the instrumentation, the measurement procedure and the measurement duration.

Optionally, there can also be provided a system controller 13, such as a computer unit, which can analyse the results delivered by the measuring means 5, 6 and possibly drive the CW-generator 8 and the laser device 1'.

In an experimental set-up made by the inventors, the laser beam (0,5 mm -6dB beam diameter) and the detector beam (2mm -6dB beam diameter), i.e. the portion of the sample surface whose emitter infrared radiation strikes the detector 11, were equicentered. This yields a stronger signal.

The modulation of the laser beam 3' is measured by a photodiode 9 connected to the amplifier 6 in parallel to the IR detector 11. The short circuit current from the photodiode 9 and from the detector 11 are assumed to be proportional to the laser intensity and the IR radiation from the surface.

Hence, masking successively the photodiode 9 and the IR detector 11, the phase of the laser modulation and that of the IR radiation are measured by the same electronic arrangement 5 and 6. It follows that the phase difference was measured without ambiguity. In this configuration the phase difference is zero for low frequencies and decreases progressively down to -45° at high frequencies.

As explained herein before, when presented in a logarithmic scale, the phase curves of various materials are obtained from a simple horizontal shift. The shift in logarithmic frequency scale expresses a ratio that is simply equal to the ratio of the thermal diffusivity of the two materials. A way to measure the diffusivity is to determine the shift that yields the best fit with the phase curve from Poco-graphite chosen as reference material.

Since the experimental set-up remained unchanged, the diameter of the laser beam and the detector sensitivity profile did not need to be known. It was only important that, for the range of frequencies used, a sufficient phase variation was obtained allowing the best fitting operation.

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As for the advantages of the present invention, it should be noted that, in comparison to the "Laser flash" method, the inventive method does not require that a small sample is taken and prepared for the measurement. It can be performed on bulk components since both heating
5 and radiative temperature measurement are performed on the same side of the sample. This is very important for many engineering applications.

Furthermore, in comparison to the "3D-photothermal radiometry", there is no need to move the sample, the laser beam or the IR detector. Another advantage is that the infrared signal is much stronger
10 since the measurement is performed directly on the heated spot. This implies that the uncertainties due to random noises are lower.

The present invention is, of course, not limited to the preferred embodiment described and represented herein, changes can be made or equivalents used without departing from the scope of the invention.

CLAIMS

1) Thermal diffusivity measurement method characterized in that it comprises the steps of:

- subjecting a surface region of the object (2), whose thermal diffusivity (α_m) has to be determined, to a modulated laser beam (3) or to the modulated beam of a similar heating source while providing a signal (MLS) thereof by, for example, measuring said beam and providing a signal proportional to the beam intensity, the heated spot or area (4) of the surface of the object (2) having a definitive diameter and a fixed intensity distribution profile;
- providing a signal (MTS) proportional to the temperature on the heated spot or area (4);
- determining the phase difference or phase shift between the modulated beam signal (MLS) and the resulting modulated temperature signal (MTS);
- using said determined phase difference or shift and its associated modulation frequency to work out the thermal diffusivity value (α_m) of said object (2).

2) Method according to claim 1, characterized in that at least one modulation parameter is subjected to controlled variation, preferably the frequency and in that the temperature is measured at the center of the heated spot or area.

3) Method according to claim 1 or 2, characterized in that the thermal diffusivity value (α_m) of the analysed object (2) is evaluated by comparison with at least one reference or reference sample whose thermal diffusivity value (α_r) has been determined previously by being subjected to the same measurement method in similar conditions.

4) Method according to claim 3, characterized in that the modulation frequency of the heating beam (3) is varied and the variation of the phase shift of the latter with respect to the heating source is recorded for a plurality of modulation frequencies, the value of the thermal diffusivity being determined by measuring the shifting required to superimpose the respective curves $\Delta\phi = F(f)$ obtained for the object (2) and a reference sample in a diagram with logarithmic frequency scale, where $\Delta\phi$ is the phase shift and f is the modulation frequency.

5) Method according to claim 3, characterized in that the phase shift between the modulated heating beam (3) and the corresponding modulated temperature is recorded for the object (2) at a given modulation frequency (f_m), then the value of the modulating frequency (f_r) yielding the same phase shift for the reference is determined and finally the value of the thermal diffusivity (α_m) of the object (2) is computed using the formula: $\alpha_m = \alpha_r \times (f_m/f_r)$, the dependence of the phase shift from the modulation frequency for the reference being known or having been previously measured.

6) Method according to claim 3, characterized in that the modulation frequency (f_m) of the heating beam (3) applied to the object (2) is varied until the phase shift between the modulated heating beam (3) and the corresponding modulated temperature reaches a predetermined value obtained previously for the reference at a given modulation frequency (f_r) and that the thermal diffusivity value (α_m) of the object (2) is computed using the formula: $\alpha_m = (\alpha_r \times f_m)/f_r$, the dependence of the phase shift from the modulation frequency for the reference being known or having been previously measured.

7) Method according to anyone of claims 1 to 6, characterized in that the modulated beam signal (MLS) and the modulated temperature signal (MTS) are both measured, successively and possibly repetitively, by measuring path and means (5, 6), preferably the same path and means for both signals, such as for example a lock-in amplifier (5) preceded by a preamplifier (6), for example by masking alternatively the beam signal generator and the temperature signal generator.

8) Method according to anyone of claims 1 to 7, characterized in that the provided heating beam (3') is modulated by means of an acousto-optical modulator (7), or a mechanical chopper, driven by an adjustable generator (8).

9) Method according to claims 7 and 8, characterized in that the modulated beam signal (MLS) is generated by an light sensor (9), such as a photodiode, receiving a deviated part of the beam, for example the light reflected by a lens (10) through which the modulated beam (3) is passing before striking the sample (2) and in that the temperature signal (MTS) is generated by an infrared sensor (11) receiving the radiation sent out by the

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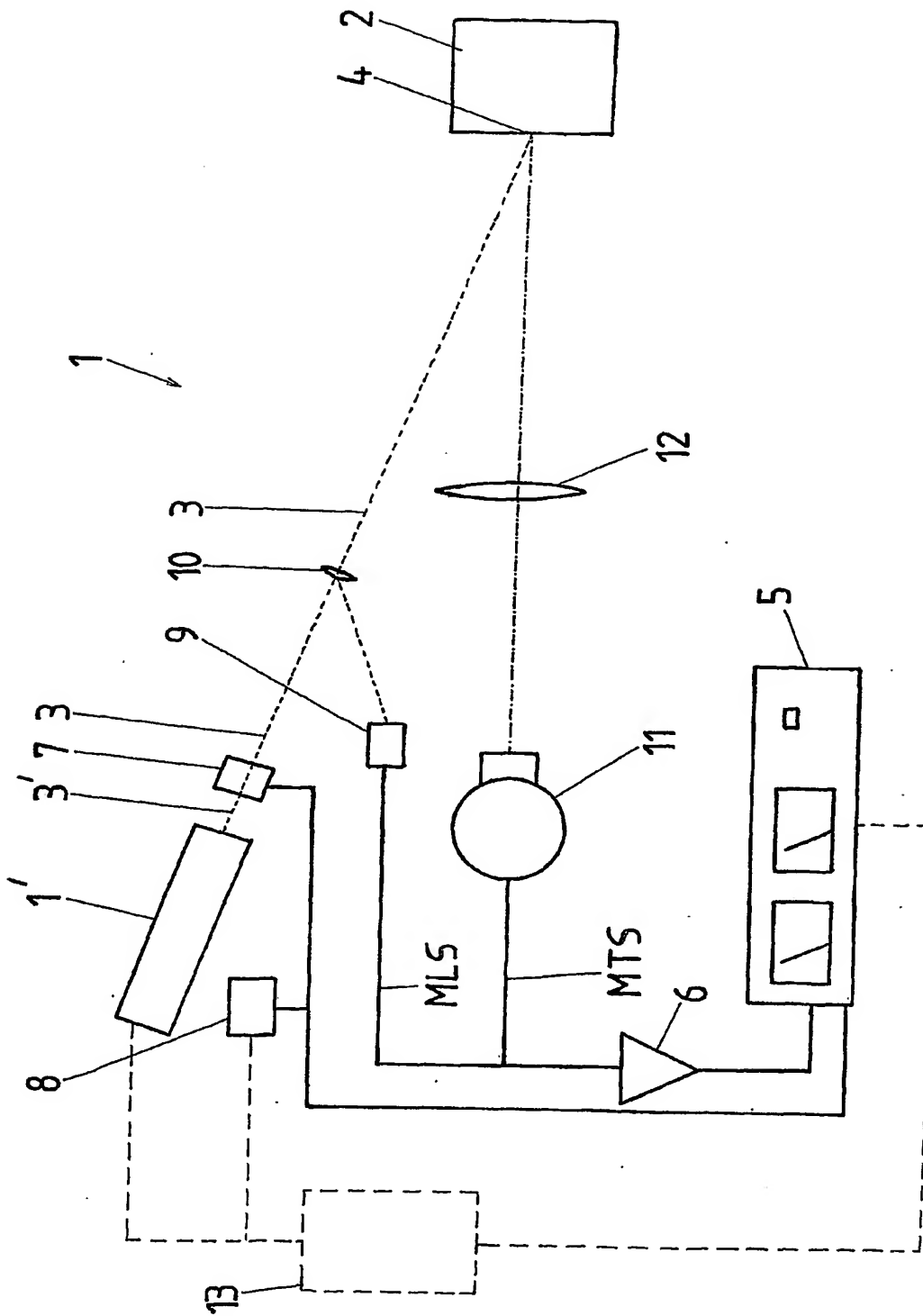
heated region or spot (4) of the surface of the analysed sample and focused by an infrared lens (12).

- 10) System for measuring the thermal diffusivity of an object, said system comprising a laser device or a similar heating source whose beam is directed towards a region or spot on the surface of said object, characterized in that said system (1) also comprises means (7, 8) for modulating said laser or heating beam (3'), means (9, 10) for generating a signal (MLS) corresponding to said modulated laser or heating beam (3), means (11, 12) for generating a signal (MTS) corresponding to the modulated temperature of the region or spot (4) struck by the modulated beam (3) and amplifying and measuring means (5, 6) able to determine at least the phase difference or shift between the modulated beam signal (MLS) and the resulting modulated temperature signal (MTS) and possibly the thermal diffusivity value (α_m) of said object (2) based upon said phase shift and associated modulation frequency.

11) System according to claim 10, characterized in that it is adapted to perform the thermal diffusivity measurement method according to anyone of claims 1 to 8.

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